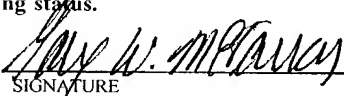


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JC07 Rec'd PCT/PTO 13 FEB 2002

FORM PTO-1390 (REV. 12-2001)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER HILL 102
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371			U.S. APPLICATION NO. (if known, see 37 CFR 1.5) <b>10/049498</b>
INTERNATIONAL APPLICATION NO. PCT/EP99/05995	INTERNATIONAL FILING DATE 16 August 1999 (16.08.99)	PRIORITY DATE CLAIMED 16 August 1999 (16.08.99)	
TITLE OF INVENTION PASSIVATION METHOD FOR ZINC-NICKEL LAYERS			
APPLICANT(S) FOR DO/EO/US Ernst-Walter Hillebrand			
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:			
<p>1. <input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</p> <p>2. <input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.</p> <p>3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.</p> <p>4. <input type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31).</p> <p>5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2))</p> <p>a. <input type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau).</p> <p>b. <input checked="" type="checkbox"/> has been communicated by the International Bureau.</p> <p>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</p> <p>6. <input checked="" type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).</p> <p>a. <input checked="" type="checkbox"/> is attached hereto</p> <p>b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4).</p> <p>7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))</p> <p>a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau).</p> <p>b. <input type="checkbox"/> have been communicated by the International Bureau.</p> <p>c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</p> <p>d. <input type="checkbox"/> have not been made and will not be made.</p> <p>8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).</p> <p>9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).</p> <p>10. <input type="checkbox"/> An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5))</p> <p><b>Items 11 to 20 below concern document(s) or information included:</b></p> <p>11. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</p> <p>12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</p> <p>13. <input checked="" type="checkbox"/> A <b>FIRST</b> preliminary amendment.</p> <p>14. <input type="checkbox"/> A <b>SECOND</b> or <b>SUBSEQUENT</b> preliminary amendment.</p> <p>15. <input type="checkbox"/> A substitute specification.</p> <p>16. <input type="checkbox"/> A change of power of attorney and/or address letter.</p> <p>17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.</p> <p>18. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4).</p> <p>19. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).</p> <p>20. <input checked="" type="checkbox"/> Other items or information: Form PCT/IB/308 is also enclosed.</p>			
EXPRESS MAIL NO. EL 845499353 US			

U.S. APPLICATION NO. <b>10/049498</b> <small>37 CFR 1.53</small>		INTERNATIONAL APPLICATION NO PCT/EP99/05995		ATTORNEY'S DOCKET NUMBER HILL 102	
21. <input checked="" type="checkbox"/> The following fees are submitted: <b>BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):</b> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO. .... <b>\$1040.00</b>  International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... <b>\$890.00</b>  International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... <b>\$740.00</b>  International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... <b>\$710.00</b>  International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) ..... <b>\$100.00</b>  <b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>				<b>CALCULATIONS PTO USE ONLY</b>	
Surcharge of <b>\$130.00</b> for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				\$ 890.00	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$	
Total claims	6 - 20 =	0	x \$18.00	\$ 0 00	
Independent claims	1 - 3 =	0	x \$84.00	\$ 0.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)				+ \$280.00	
<b>TOTAL OF ABOVE CALCULATIONS =</b>				\$ 890.00	
<input checked="" type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.				\$ 445.00	
<b>SUBTOTAL =</b>				\$	
Processing fee of <b>\$130.00</b> for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				\$	
<b>TOTAL NATIONAL FEE =</b>				\$	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). <b>\$40.00</b> per property +				\$	
<b>TOTAL FEES ENCLOSED =</b>				\$ 445.00	
				Amount to be refunded:	\$
				charged:	\$
a. <input checked="" type="checkbox"/> A check in the amount of \$ <u>445.00</u> to cover the above fees is enclosed.  b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.  c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>50/1039</u> . A duplicate copy of this sheet is enclosed.  d. <input type="checkbox"/> Fees are to be charged to a credit card. <b>WARNING:</b> Information on this form may become public. <b>Credit card</b> <b>information should not be included on this form.</b> Provide credit card information and authorization on PTO-2038.					
<b>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.</b>  SEND ALL CORRESPONDENCE TO Gary W. McFarron Cook, Alex, McFarron, Manzo, Cummings & Mehler, Ltd. 200 West Adams Street - Suite 2850 Chicago, IL 60606 (312) 236-8500					
				 SIGNATURE Gary W. McFarron NAME <u>27,357</u> REGISTRATION NUMBER	

PATENT  
Attorney Docket No. HILL 102

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:	)	<b>CERTIFICATE OF MAILING</b>
Ernst-Walter Hillebrand	)	<b>BY "EXPRESS MAIL"</b>
International Application No.:	)	Express Mailing Label : <u>EL845499353US</u>
PCT/EP99/05995	)	Date of Deposit <u>February 13, 2002</u>
International Application Filing Date:	)	I hereby certify that this paper or fee is being
August 16, 1999	)	deposited with the United States Postal
International Priority Date:	)	Service Express Mail Post Office Box
August 16, 1999	)	Addressee" service under 37 CFR 1.10 on
For: PASSIVATION METHOD FOR ZINC-	)	the date indicated above and is addressed
NICKEL LAYERS	)	to: Box PCT, Commissioner for Patents,
	)	Washington, D.C. 20231
	)	NAME <u>ARMANDO CHING</u>
	)	(TYPED OR PRINTED)
	)	SIGNATURE <u>Armando Ching</u>
	)	DATE: <u>February 13, 2002</u>

Commissioner for Patents  
Washington, D.C. 20231

PRELIMINARY AMENDMENT

Dear Sir/Madam:

Please enter this Preliminary Amendment prior to examination  
and calculation of the filing fee.

IN THE CLAIMS:

Please amend Claim 3 as follows:

3 (Amended). The method of claim 1 characterized in that a  
conversion coat is applied to the zinc/nickel coat.

5 (Amended). The method of claim 1 characterized in that a  
coat of dry lubricant is applied.

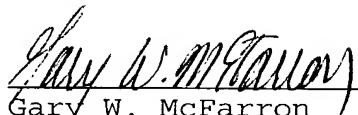
Please add new claim 6 as follows:

6 (New). The method of claim 2 characterized in that the zinc/nickel coat is oxidized at pH 1.8.

REMARKS

This is a Preliminary Amendment to the above-identified patent application. In line 1 of claim 3, "or 2," has been deleted and has been replaced with --claim 1--. In line 1 of claim 5, "one of the preceding claims," has been deleted and has been replaced with --claim 1--. These amendments are made to remove multiple dependencies in Claims 3 and 5. In addition, new claim 6 has been added.

Respectfully submitted,



Gary W. McFarron  
Registration No.: 27,357

COOK, ALEX, MCFARRON, MANZO  
CUMMINGS & MEHLER, LTD.  
200 West Adams Street, Suite 2850  
Chicago, Illinois 60606  
(312)236-8500

VERSION WITH MARKINGS TO SHOW CHANGES MADE

3 (Amended). The method of claim 1 ~~or 2~~, characterized in that a conversion coat is applied to the zinc/nickel coat.

5 (Amended). The method of ~~one of the preceding claims~~ claim 1~~7~~, characterized in that a coat of dry lubricant is applied.

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## PASSIVATION METHOD FOR ZINC-NICKEL COATS

The invention relates to a method of passivating zinc-nickel coats.

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The passivation of electroplate coatings is known and serves for corrosion protection and also as a tie substrate for further coatings, such as plastic coatings or paints, for example.

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The prior art methods fall back on a chromating operation, in the course of which, preferably, a chromium(VI) coat is produced which provides good corrosion resistance. Here, in conjunction with zinc, blue-yellow, black and olive chromate conversion coats and, for nickel transparent, yellow and black chromate conversion coats are known, each of which differ in their corrosion resistance.

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For the zinc-nickel field, black chromating as corrosion protection with a preferential esthetic effect has found widespread use.

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The German laid-open specification 33 02 502 describes a chromating method for a zinc-cobalt coating.

25

The widespread use of chromates as a corrosion protection coat possesses considerable disadvantages. For instance, the chromium(VI) employed primarily is carcinogenic. An additional protective coating is therefore necessary in order to prevent skin contact. This leaves unresolved, however, the problem that chromium(VI)-coated parts constitute a considerable environmental burden, particularly as disused contaminated material. The costs of environment-friendly disposal of chromium(VI)-coated parts are high.

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In order to avoid the unwanted chromium(VI), it is also possible to employ chromium(III) passivation with a blue color. However, like the other known alternative of molybdenum passivation, the chromium(III) passivation possesses inadequate corrosion protection properties. In particular, the two aforementioned chromating methods are not suitable for zinc-nickel coatings.

A further problem which occurs primarily with the black passivation of zinc-nickel coatings lies in the approximately 2  $\mu\text{m}$  of material removed from the zinc-nickel coat. At a total coat thickness of about 10  $\mu\text{m}$ , this removed material represents a cost factor of about 20%.

Additionally, the rise of chromium(III) and zinc in the chromating solution results in this solution being rapidly consumed, and necessitates frequent rebatching of the solution and disposal of the spent solution.

The invention is therefore based on the problem of providing a passivation for zinc-nickel coatings which is not accompanied by any health hazard nor by difficulties associated with disposal, and which leads to a cost saving.

The problem is solved by a method as claimed in claim 1.

In this method, the zinc-nickel surface is treated with an oxidizing agent, avoiding any use of chromium, and can subsequently be coated with a further coat.

The coating can serve to improve the visual quality of the surface or to increase the slip properties. Furthermore, other coats can be applied as a corrosion protection coat.

A particular advantage of the passivation of the invention is its good red rust resistance. This is attributable to the surface structure which arises from the oxidative treatment.

5

The zinc-nickel coat passivated in accordance with the invention can be treated with any desired conversion coat or else directly with a low-friction lacquer. Suitable conversion coats include organic or inorganic coating systems: silicates or polymer waxes, for example.

The conversion coat is preferably composed of Aquares, which in this combination affords particular protection against white rust. Atop the Aquares coat it is then possible, additionally, to apply a low-friction lacquer in order to achieve optimum slip properties in the coated component. A preferred low-friction lacquer used is Molykote D708 from the company Molykote.

20

In the text below, an exemplary embodiment is described in greater detail in order to illustrate the invention.

The components are first of all electrocoated with a 12 to 15.5% zinc-nickel coat. This zinc-nickel coat is oxidized using ammonium peroxide sulfate at a pH of 1.8. In order to improve the visual or technical quality, the oxidized zinc-nickel coat is aftertreated. This aftertreatment may consist of an inorganic or organic film.

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Example 1: (inorganic film)

An inorganic film is formed by a solution containing sodium silicate in dissolved form:

35

50 g/l sodium silicate  
pH of 8-10 (set using sodium hydroxide solution or dilute phosphoric acid)

40



Example 2: (organic film)

50 g/l acrylate-styrene copolymer (such as Acronal  
567 D from BASF)  
5 2 g/l isopropanol  
0.01 g/l thickener  
pH 8-10 (set using dilute ammonia)

Example 3: (organic film)

10 25 g/l polyethylene wax (such as Luwax OA2 from BASF)  
2 g/l Lutensol ON110 (surfactant, BASF, as emulsifier  
for wax)  
pH = 8-10 (set using dilute ammonia)

15

Example 4: (organic film)

It is also possible to apply organic films by means of  
electrocoating. Suitable with preference for this  
20 purpose is a cathodic dip coating operation in which  
the workpiece is connected as the cathode in a  
corresponding aqueous solution. Hydrogen is formed at  
the cathode and, consequently, there is an increase in  
the pH in the cathode film. At high pH, the dissolved  
25 organic constituents are precipitated and form a thin  
film on the surface. This film greatly reduces the  
surface conductivity. When all of the surface has been  
coated, therefore, there is a considerable increase in  
voltage and the coating process is at an end.  
30 Downstream drying is then a baking operation at  
approximately 180°C.

In appropriate solutions, furthermore, it is also  
possible to connect the workpieces as the anode (anodic  
35 electrocoating). In this case, oxygen is evolved at the  
anode and hence the pH is lowered downward (lower  
values). The polycarboxylic acids dissolved beforehand,  
for example, with ammonia are then deposited again.

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## Claims

1. A method of passivating galvanic zinc/nickel  
5 coatings characterized in that the coating is treated  
with an oxidizing agent.
2. The method of claim 1, characterized in that a  
peroxide sulfate is used as oxidizing agent.
- 10 3. The method of claim 1 or 2, characterized in that  
a conversion coat is applied to the zinc/nickel coat.
4. The method of claim 3, characterized in that the  
15 conversion coat is composed of a polymer wax.
5. The method of one of the preceding claims,  
characterized in that a coat of dry lubricant is  
applied.

(12) NACH DEM VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES  
PATENTWESENS (PCT) VERÖFFENTLICHTE INTERNATIONALE ANMELDUNG(19) Weltorganisation für geistiges Eigentum  
Internationales Büro(43) Internationales Veröffentlichungsdatum  
22. Februar 2001 (22.02.2001)

PCT

(10) Internationale Veröffentlichungsnummer  
**WO 01/12877 A1**(51) Internationale Patentklassifikation<sup>7</sup>: C23C 22/53

(21) Internationales Aktenzeichen: PCT/EP99/05995

(22) Internationales Anmeldedatum:  
16. August 1999 (16.08.1999)

(25) Einreichungssprache: Deutsch

(26) Veröffentlichungssprache: Deutsch

(71) Anmelder (für alle Bestimmungsstaaten mit Ausnahme von US): **WALTER HILLEBRAND GMBH & CO. GALVANOTECHNIK** [DE/DE]; Westerhaar 56-58, 58739 Wickede/Ruhr (DE).

(72) Erfinder; und

(75) Erfinder/Anmelder (nur für US): **HILLEBRAND, Ernst-Walter** [DE/DE]; Am Brauck 19, D-58739 Wickede/Ruhr (DE). *DEX*(74) Anwälte: **KÖNIG, Reimar** usw.; König Palgen Schumacher Kluin, Lohengrinstrasse 11, D-40549 Düsseldorf (DE).

(81) Bestimmungsstaaten (national): BR, CA, CN, CZ, EE, HU, IL, JP, KP, MX, NO, PL, SK, TR, US.

(84) Bestimmungsstaaten (regional): europäisches Patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Veröffentlicht:

— Mit internationalem Recherchenbericht.

Zur Erklärung der Zweibuchstaben-Codes, und der anderen Abkürzungen wird auf die Erklärungen ("Guidance Notes on Codes and Abbreviations") am Anfang jeder regulären Ausgabe der PCT-Gazette verwiesen.

(54) Title: PASSIVATION METHOD FOR ZINC-NICKEL LAYERS

(54) Bezeichnung: PASSIVIERUNGSVERFAHREN FÜR ZINK-NICKEL-SCHICHTEN

(57) Abstract: The invention relates to a method for the passivation of electrodeposited zinc-nickel coatings, according to which the coating is treated with an oxidizing agent, thus obviating the need for chromium-VI.

(57) Zusammenfassung: Die Erfindung betrifft ein Verfahren zur Passivierung von galvanischen Zink/Nickel-Überzügen, bei denen der Überzug mit einem Oxidationsmittel behandelt wird, wodurch sich die Verwendung von Chrom-VI vermeiden läßt.

Please type a plus sign (+) inside this box → ☐

PTO/SB/01 (10-00)

Approved for use through 10/31/2002. OMB 0851-0032

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

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<b>DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION</b> <b>(37 CFR 1.63)</b>  <input type="checkbox"/> Declaration Submitted with Initial Filing      OR <input type="checkbox"/> Declaration Submitted after Initial Filing (surcharge (37 CFR 1.16 (e)) required)	<b>Attorney Docket Number</b>	HILL 102
	<b>First Named Inventor</b>	Ernst-Walter Hillebrand
	<b>COMPLETE IF KNOWN</b>	
	<b>Application Number</b>	/
	<b>Filing Date</b>	Herewith
	<b>Group Art Unit</b>	
	<b>Examiner Name</b>	

As a below named inventor, I hereby declare that:

My residence, mailing address, and citizenship are as stated below next to my name

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

PASSIVATION METHOD FOR ZINC-NICKEL LAYERS

(Title of the invention)

the specification of which

☐ is attached hereto

OR

☒ was filed on (MM/DD/YYYY)

08/16/1999

as United States Application Number or PCT International

Application Number

PCT/EP99/05995

and was amended on (MM/DD/YYYY)

(if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the national or PCT international filing date of the continuation-in-part application.

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached?	
				YES	NO
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

☐ Additional foreign application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto:

I hereby claim the benefit under 35 U.S.C. 119(e) of any United States provisional application(s) listed below.

Application Number(s)	Filing Date (MM/DD/YYYY)	<input type="checkbox"/> Additional provisional application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

[Page 1 of 2]

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Approved for use through 10/31/2002 OMB 0651-0032

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NAME OF SOLE OR FIRST INVENTOR :

☐ A petition has been filed for this unsigned inventor

Given Name

(first and middle (if any)) Ernst-Walter

Family Name

or Surname Hillebrand

Inventor's  
Signature 

Date 15.02.2002

Residence: City 58739 Wickede

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NAME OF SECOND INVENTOR:

☐ A petition has been filed for this unsigned inventor

Given Name

(first and middle (if any))

Family Name  
or SurnameInventor's  
Signature

Date

Residence: City

State

Country

Citizenship

Mailing Address

Mailing Address

City

State

ZIP

Country

☐ Additional inventors are being named on the \_\_\_\_\_ supplemental Additional Inventor(s) sheet(s) PTO/SB/02A attached hereto.